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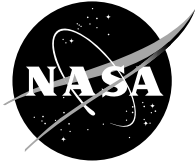
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ULTIMATE TENSILE STRENGTH AS A FUNCTION OF TEST RATE FOR VARIOUS CERAMIC MATRIX COMPOSITES AT ELEVATED TEMPERATURES

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Ultimate tensile strength of five different continuous fiber-reinforced ceramic composites, including SiC/BSAS (2D, 2 types), SiC/MAS-5 (2D), SiC/SiC (2D, enhanced) and C/SiC (2D), was determined as a function of test rate at 1100 to 1200 °C in air. All five composite materials exhibited a significant dependency of ultimate strength on test rate such that the ultimate strength decreased with decreasing test rate, similar to the behavior observed in many advanced monolithic ceramics at elevated temperatures. The application of the preloading technique as well as the prediction of life from one loading configuration (constant stress-rate) to another (constant stress loading) for SiC/BSAS suggested that the overall macroscopic failure mechanism of the composites would be the one governed by a power-law type of damage evolution/accumulation, analogous to slow crack growth commonly observed in advanced monolithic ceramics.

1. INTRODUCTION

The successful development and design of continuous fiber-reinforced ceramic composites (CFCCs) are dependent on thorough understanding of basic properties such as fracture and delayed failure (slow crack growth, fatigue, or damage accumulation) behavior. Particularly, complete evaluation of delayed failure behavior under specified loading/environment conditions is a prerequisite to ensure accurate life prediction of structural components.

This paper, as a continuation of the previous studies,^{1,2} describes the effect of test rate on elevated-temperature ultimate tensile strength of five different fiber-reinforced ceramic matrix composites including 2D SiC_f/BSAS (2 types), 2D SiC_f/MAS-5, 2D SiC_f/SiC (enhanced) and C_f/SiC composites. For each composite, ultimate tensile strength was determined in air as a function of test rate at elevated temperature of 1100 °C (for SiC/BSAS and SiC/MAS-5) or 1200 °C (for SiC/SiC and C/SiC). This type of testing, when used for monolithic ceramics, is called “constant stress-rate” or “dynamic fatigue” testing.³⁻⁵ The test rate dependency of ultimate strength was analyzed with the power-law (damage) propagation, conventionally utilized for monolithic ceramics and glass. Preloading tests were conducted to better understand the governing failure mechanism(s) of the composites. Finally, the result of elevated-temperature constant stress (“static fatigue” or “stress rupture”) testing was obtained for SiC/BSAS and compared with that of constant stress rate testing. This was done to verify the overall failure mechanism(s) of the composites and to establish constant stress-rate testing as a means of life prediction test methodology for CFCCs.

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2. EXPERIMENTAL PROCEDURE

The five ceramic matrix composites tested in this study were Nicalon™ (SiC) fiber-reinforced barium strontium aluminosilicate (designated Nicalon/BSAS, 2D crossply), Hi-Nicalon™ fiber-reinforced BSAS (designated Hi-Nicalon/BSAS, 2D crossply), Nicalon fiber-reinforced magnesium aluminosilicate (designated SiC/MAS-5, 1D), Nicalon fiber-reinforced silicon carbide (designated SiC/SiC, 2D woven, enhanced), and carbon fiber-reinforced silicon carbide matrix (designated C/SiC, 2D woven) composites. The matrices of the composites were reinforced by (Nicalon, Hi-Nicalon, or carbon) fibers with a fiber volume fraction of around 0.39 to 0.42. The crossply or plain-woven laminates of the composites were typically 12 to 18 plies thick with a nominal thickness of around 3 to 3.5 mm, depending on material. Both Nicalon/BSAS and Hi-Nicalon/BSAS were fabricated at NASA/GRC,⁶ SiC/MAS-5 by Corning, SiC/SiC by DuPont Company, and C/SiC by Honeywell Advanced Composites.⁷ Information regarding the composite materials and processing can be found elsewhere.⁶⁻⁸ The dogboned tensile test specimens measuring 152.4 mm (length) x 12.7 mm (width) were machined from the composite laminates, with the gage section of about 30 mm long, 10 mm wide and 3.0 to 3.5 mm thick (as-furnished). The C/SiC composites were supplied with a notch machined (in depth=2.5 mm and root radius=1.2 mm) at the longitudinal center of each test specimen.

Monotonic tensile testing was conducted in ambient air at 1100 °C for Nicalon/BSAS, Hi-Nicalon/BSAS and SiC/MAS-5 and at 1200 °C for SiC/SiC and C/SiC, using a servohydraulic test frame (Model 8501, Instron, Canton, MA). A total of three different test rates (in load control), corresponding to stress rates of 5, 0.158, and 0.005 MPa/s, were employed with typically one to three test specimens tested at each test rate. Heating of test specimens was conducted using an induction-heating SiC susceptor system.⁸ Two high-temperature extensometers were placed on edges of each test specimen to measure tensile strain. Preloading or accelerated testing, primarily applied to monolithic ceramics⁹ to save test time, was also conducted at test temperatures using 0.005 MPa/s in an attempt to better understand the governing failure mechanism of the materials. Predetermined preloads, corresponding to 80 % of the ultimate strength determined at 0.005 MPa/s with no preload, were applied quickly to the test specimens prior to testing and their corresponding ultimate strengths were determined. One test specimen was used in preload testing. Tensile testing was performed in accordance with ASTM Test Standard, ASTM C 1359.¹⁰

Constant stress (“static fatigue” or “stress rupture”) tensile testing was also performed at 1100 °C in ambient air for the Nicalon/BSAS composite (for both batches ‘A’ and ‘B’) using test specimens with the same geometry and the same test frame that were used for monotonic tensile testing. The limited availability of test materials confined the testing to three to four test specimens, depending on batch. Two to three different applied static loads were applied to test specimens and their corresponding times to failure were determined.

3. RESULTS AND DISCUSSION

3.1. Ultimate Tensile Strength (Constant Stress-Rate Testing)

The results of monotonic tensile strength testing for five different composites with different test rates are presented in Figure 1, where *ultimate tensile strength* was plotted as a function of *applied stress rate* using log-log scales. Each solid line in the figure indicates a best-fit regression line based on the log (*ultimate strength*) versus log (*applied stress rate*) relation. The decrease in ultimate tensile strength with decreasing stress rate, which represents a susceptibility to damage accumulation or delayed failure, was significant for all the composite materials, consistent with the results of the previous study.^{1,2} The strength degradation was about 43, 59, 43, 48, and 28 %, respectively, for Nicalon/BSAS, Hi-Nicalon/BSAS, SiC/MAS-5, SiC/SiC and C/SiC, when stress rate decreased from the highest (5 MPa/s) to the lowest

(0.005 MPa/s). Fracture patterns for the SiC/MAS-5 composite indicated some typical fiber pullout with jagged faceted matrix cracking often propagating along the test-specimen length. The mode of fracture for both Nicalon/BSAS and Hi-Nicalon/BSAS composites also showed fiber pullout with jagged matrix cracking through the specimen-thickness direction. No significant difference in the mode of fracture was observed for SiC/SiC and C/SiC, where almost all the specimens tested at either high or low stress rate exhibited relatively flat fracture surfaces (see Figure 2), called *brittle* fracture.

The strength dependency on test rate exhibited by the five composites (Figure 1) is very similar to that observed in advanced monolithic ceramics (or brittle materials including glass and glass ceramics) at ambient or elevated temperature. The strength degradation with decreasing stress rate in brittle materials is known to occur by slow crack growth (delayed failure or fatigue) of an initial crack, and is expressed as follows³⁻⁵

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \quad (1)$$

where n and D are slow crack growth (SCG) parameters, and σ_f (MPa) and $\dot{\sigma}$ (MPa/s) are strength and stress rate, respectively. This equation is based on the conventional power-law crack velocity formulation as expressed

$$v = A(K_I / K_{IC})^n \quad (2)$$

where v , K_I and K_{IC} are crack velocity, mode I stress intensity factor and fracture toughness, respectively. A is also called SCG parameter.

Constant stress-rate (“dynamic fatigue”) testing based on Equation (1) has been established as ASTM Test Methods (C1368² and C1465³) to determine SCG parameters of advanced monolithic ceramics at ambient and elevated temperatures. Notwithstanding the limited number of test specimens used, the data fit to Equation (1) was very reasonable with the coefficients of correlation (r_{coef}) all greater than 0.930. This implies that damage evolution/accumulation or delayed failure of the composites would be adequately described by a power-law type relation, Equation (2). With this assumption, the *apparent* parameters n' and D' for the composites were determined using a linear regression analysis based on Equation (1) with the experimental data in Figure 1: $n' = 13$ and $D' = 127$, $n' = 7$ and $D' = 188$, $n' = 13$ and $D' = 367$, $n' = 20$ and $D' = 160$, and $n' = 18$ and $D' = 166$, respectively, for Nicalon/BSAS, Hi-Nicalon/BSAS, SiC/MAS-5, SiC/SiC, and C/SiC (The prime was used here for composites to distinguish them from monolithic ceramic counterparts.). It is noteworthy that the value of n' , a measure of susceptibility to damage, was very low for Nicalon/BSAS, Hi-Nicalon/BSAS and SiC/MAS-5, and intermediate for SiC/SiC and C/SiC. Similar results were also found from the previous study^{1,2} with SiC/CAS (1D), SiC/MAS (2D) and SiC/SiC (2D, ‘standard’) composites. Note that monolithic silicon nitrides typically exhibit $n \geq 20$ at temperatures ≥ 1200 °C.

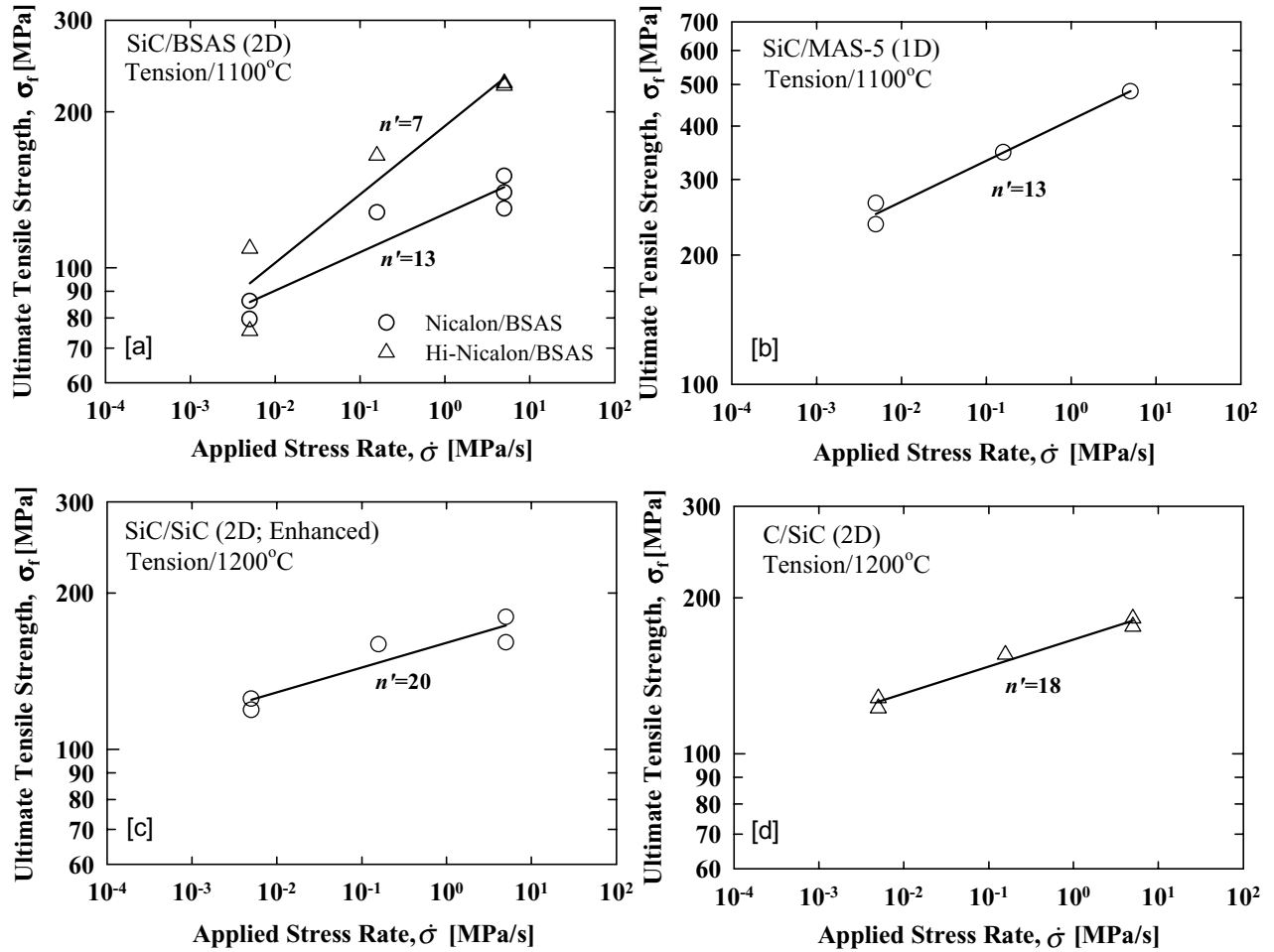


Figure 1. Results of ultimate tensile strength as a function of applied stress rate determined for (a) Nicalon/BSAS and Hi-Nicalon/BSAS, (b) SiC/MAS-5, (c) SiC/SiC and (d) C/SiC ceramic matrix composites at elevated temperatures in air. The solid lines represent the best-fit regression lines based on Equation (1).

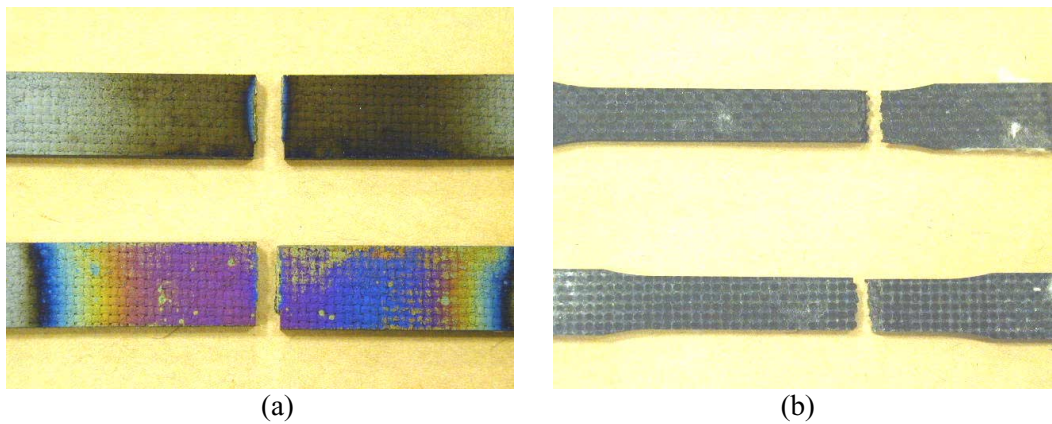


Figure 2. Fracture patterns for (a) SiC/SiC and (b) C/SiC showing 'brittle' fracture behavior. The upper and lower pictures for a given composite material indicate the specimens tested at the lowest and the highest test rates, respectively.

3.2. Preload Testing

The results of preloading tests conducted at 0.005 MPa/s are depicted in Figure 3, where the ultimate strength was plotted against ‘preloading factor’ (α) for each composite. The preloading factor α is defined such that a preload stress applied to the test specimen is normalized with respect to the strength with no preload. Each solid line in Figure 3 indicates the theoretical prediction of ultimate strength as a function of preload with the estimated value of n' and applied α ($=0.8$) together with the strength data with no preload, based on the following equation⁹

$$\sigma_{fp} = \sigma_f \left(1 + \alpha^{n+1}\right)^{\frac{1}{n+1}} \quad (3)$$

where σ_{fp} is strength with a preload, σ_f is strength with no preload (regular testing), and α is within $0 \leq \alpha < 1$. The prediction, despite a limited number of test specimens used, is in good agreement with the experimental data except for SiC/MAS-5, as seen in the figure. Note that Equation (3) was derived based on the power-law SCG relation of Equation (2). Therefore, the applicability of the preloading analysis to

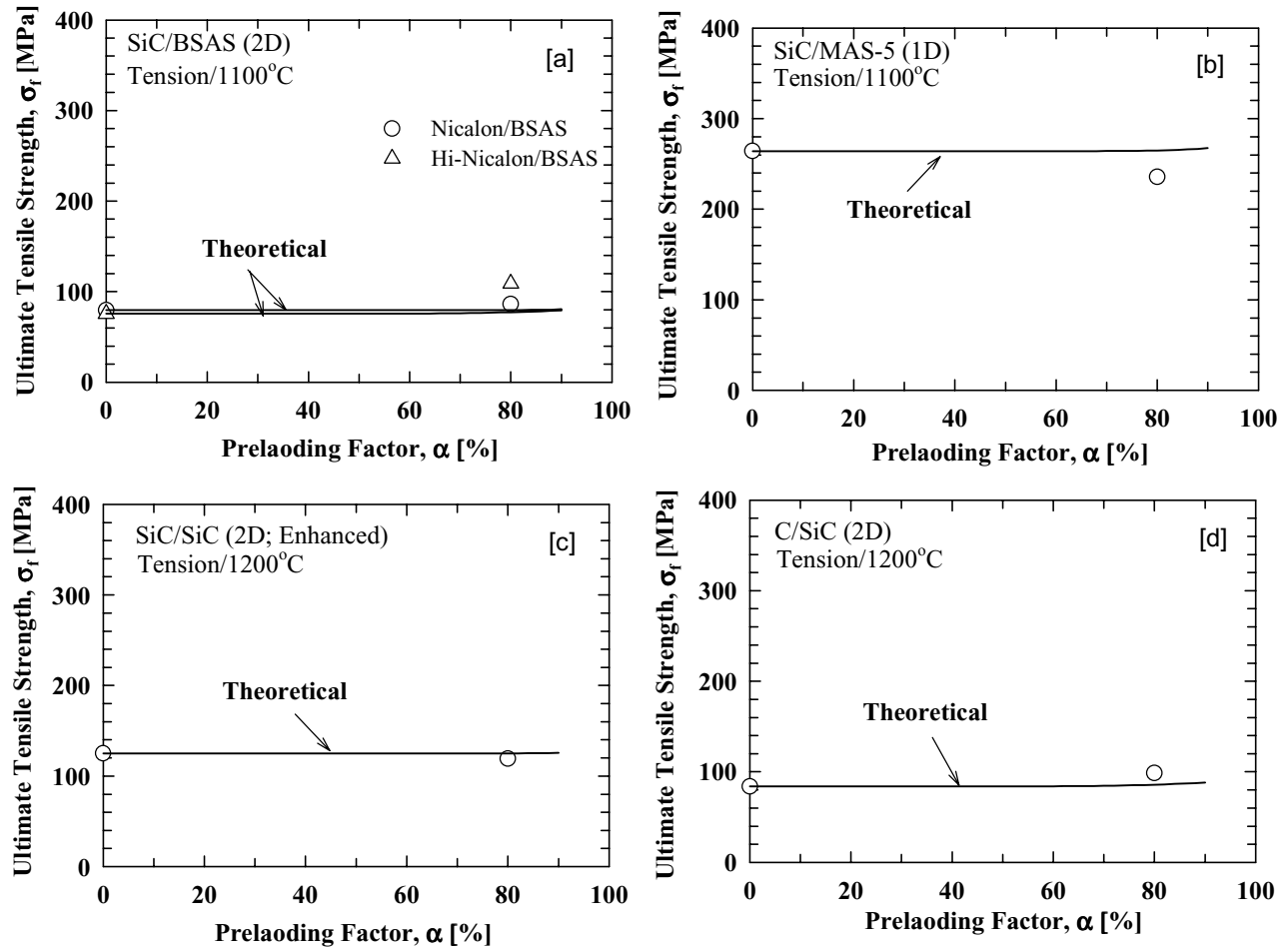


Figure 3. Result of preloading tests (ultimate tensile strength as a function of preloading) for (a) Nicalon/BSAS and Hi-Nicalon/BSAS, (b) SiC/MAS-5, (c) SiC/SiC and (d) C/SiC ceramic matrix composites at elevated temperatures in air. A theoretical prediction based on Equation (3)⁹ is included for comparison for each composite material.

the composite materials suggests that major damage evolution/accumulation process of the composites leading to failure would be the one governed by the power-law relation, which is consistent with the observation of the previous preload study for other ceramic matrix composites.² It is also noted that the overall difference in ultimate strength between two preloads (0 and 80 %) was insignificant. This indicates that any significant damage that would control ultimate strength of the material did not occur before the applied load up to 80 % of fracture load. Conversely, the damage to control final failure would have occurred when applied load or test time was greater than 80 % of fracture load or total test time. The detailed analysis and application of the preloading technique for brittle (monolithic) materials can be found elsewhere.⁹

3.3. Constant Stress (“Stress Rupture”) Testing

A summary of results of constant stress testing for Nicalon/BSAS (with two different batches ‘A’ and ‘B’) at 1100 °C is presented in Figure 4, where *time to failure* was plotted against *applied stress* in log-log scales. A decrease in time to failure with increasing applied stress, which represents a susceptibility to damage accumulation or delayed failure, was evident for the composite. The mode of fracture in constant stress testing was similar to that in constant stress-rate testing showing some fiber pullout with jagged matrix cracking through the specimen-thickness direction. The solid lines in Figure 4 indicate life prediction from the constant stress-rate data (Note that the constant stress-rate data for batch ‘B’ was not presented in Figure 1 for clarity of data presentation). The prediction, primarily applied for brittle (monolithic) materials, was made using the following equation based on the power-law SCG formulation of Equation (2):¹¹

$$t_f = \left[\frac{D^{n+1}}{n+1} \right] \sigma^{-n} \quad (4)$$

where t_f and σ are time to failure and applied stress, respectively. Use of Equation (4) together with n' and D' determined in constant stress-rate testing allows one to predict life in constant stress loading. The result thus predicted shows a reasonable agreement with experimental data at least for short periods of life (see Figure 4). Hence it can be stated that the governing failure mechanism of Nicalon/BSAS is almost identical in either constant stress-rate testing or constant stress testing. Since the prediction (Equation (4)) was made based on the power-law relation, it is certain that the governing failure mechanism of Nicalon/BSAS would be the one controlled by the power-law type of damage evolution/accumulation or delayed failure. The results shown in Figure 3 are also consistent with the previous study with other ceramic matrix composites.²

Finally, the results of this work suggest that care must be exercised when characterizing elevated-temperature ultimate strength of composite materials. This is due to that fact that elevated-temperature ultimate strength has a relative meaning if a material exhibits rate dependency: the strength simply depends on which test rate one chooses (Figure 1). Therefore, at least two test rates (high and low) are recommended to better characterize the high-temperature ultimate strength behavior of a composite material.

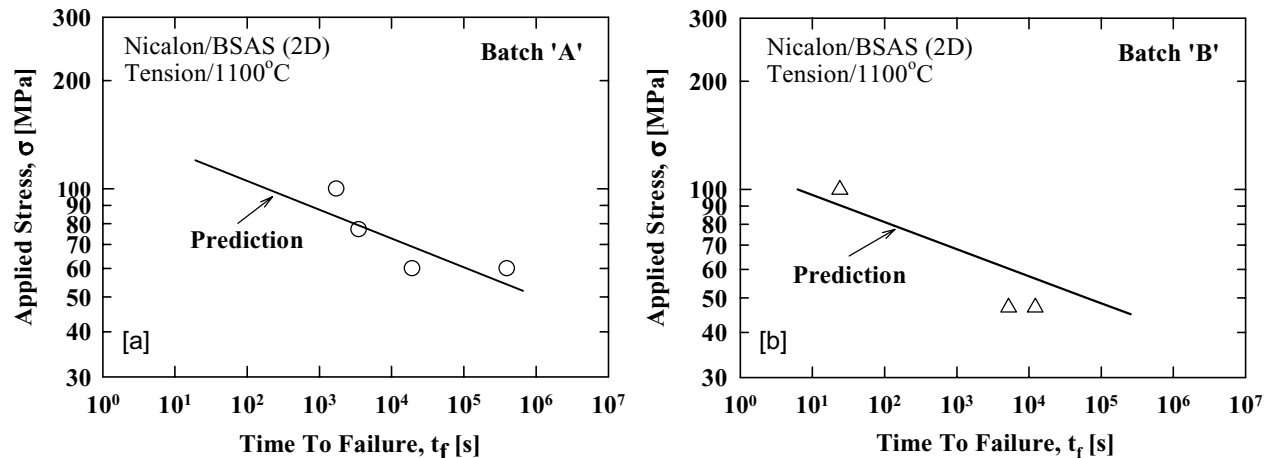


Figure 4. Results of constant stress (“static fatigue” or “stress rupture”) testing for Nicalon/BSAS composite: (a) Batch ‘A’ and (b) Batch ‘B’” tested at 1100 °C in air. The solid lines represent the predictions made based on the results of constant stress-rate testing (Figure 1).

4. CONCLUSIONS

Elevated-temperature ultimate tensile strength of five different continuous fiber-reinforced ceramic composites, including Nicalon/BSAS, Hi-Nicalon/BSAS, SiC/MAS-5, SiC/SiC, and C/SiC, exhibited a strong dependency on test rate, consistent with the behavior observed in other ceramic matrix composites as well as in many advanced monolithic ceramics at elevated temperatures. The applicability of the preloading technique and the predictability of life from one loading configuration (constant stress-rate) to another (constant stress loading) suggested that the overall failure mechanism of the composite materials would be a process primarily governed by a power-law type of damage evolution/accumulation. It was further found that constant stress-rate testing could be utilized as a means of life prediction test methodology even for composites when short lifetimes are expected.

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